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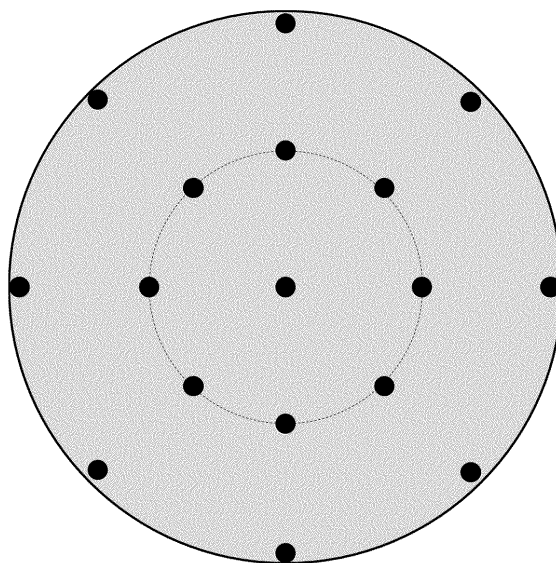
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(54) **TUNGSTEN-SINTERED-BODY SPUTTERING TARGET AND METHOD FOR PRODUCING SAME**

(57) Provided is a tungsten sintered compact sputtering target containing iron as an impurity in an amount of 0.8 wtppm or less, and remainder being tungsten and other unavoidable impurities, wherein a range of iron concentration in a target structure is within a range of  $\pm 0.1$  wtppm of an average concentration. Additionally provided is a tungsten sintered compact sputtering target according to claim 1 or claim 2, wherein a relative density of the target is 99% or higher, an average crystal grain size is 50  $\mu\text{m}$  or less, and a crystal grain size range is 5 to 200  $\mu\text{m}$ . The present invention aims to inhibit abnormal grain growth in the tungsten target by reducing the amount of iron in the tungsten sintered compact sputtering target.

[Fig. 1]



**Description**

## TECHNICAL FIELD

5 **[0001]** The present invention relates to a tungsten sintered compact target that is used upon forming, via the sputtering method, a gate electrode or a wiring material of an IC, LSI or the like, and to a method of producing such a tungsten sintered compact target.

## BACKGROUND ART

10 **[0002]** In recent years, pursuant to the higher integration of very-large-scale integrated circuits ("VLSI"), studies are being conducted for using materials having lower electrical resistivity as the electrode material or the wiring material. Under the foregoing circumstances, high-purity tungsten having low resistivity and stable thermal and chemical characteristics is being used as the electrode material or the wiring material.

15 The foregoing electrode material or wiring material for VLSI is generally produced by way of the sputtering method or the CVD method, but the sputtering method is being widely used in comparison to the CVD method since the structure and operation of the equipment are relatively simple, deposition can be performed easily, and the process is of low cost.

**[0003]** While a tungsten target is demanded of high purity and high density, in recent years, as an electrode material or a wiring material for VLSI, a material with even lower electrical resistivity is being demanded in a film deposited by sputtering a tungsten target.

20 **[0004]** As described later, a tungsten sintered compact target is capable of attaining higher purity and high densification, and, while there are disclosures for achieving such higher purity and high densification, research and development regarding the abnormal grain growth of tungsten and deterioration in the target strength have not been conducted.

**[0005]** Conventionally, a tungsten sintered compact sputtering target was produced via pressure sintering using a graphite die. For example, this conventional method is described in Patent Document 1, Patent Document 2, and Patent Document 3. In the foregoing case, there is a possibility that C, as an impurity, will inevitably get mixed into tungsten. Moreover, while the type of die used is not particularly specified, Patent Document 4 and Patent Document 5 describe methods of achieving higher density.

The foregoing Patent Documents mainly aim to achieve the higher density of a tungsten target.

30 **[0006]** Otherwise, Patent Document 6 describes lowering the C content in a tungsten sintered compact target, and specifically discloses reducing the carbon content to be 50 ppm or less (the most reduced C content was 19 ppm in the Examples).

**[0007]** Moreover, Patent Document 7 discloses a technique of reducing the C content in a metal material (the most reduced C content was 10 ppm in the Examples) aiming to achieve the uniformity of the film and reduction of dust generation.

35 Moreover, Patent Document 8 discloses a technique of causing the C content to be 30 ppm or less (the most reduced C content was 6 ppm in the Examples) for producing a high-purity and high-density tungsten sintered compact target.

**[0008]** Meanwhile, it was discovered that there is a problem in that abnormal grain growth and deterioration in target strength occur during the production stage of a tungsten sintered compact sputtering target and consequently deteriorate the product yield. As a method of resolving this problem, the present Applicant discovered that the inclusion of phosphorus significantly affects the abnormal grain growth of tungsten and deterioration in target strength, and proposed causing the phosphorus contained in the tungsten to be 1 ppm or less as described in Patent Document 3.

Consequently, it became possible to prevent the abnormal grain growth of tungsten and improve the target product yield, and at that stage Patent Document 3 was an extremely effective method.

45 **[0009]** Nevertheless, while it was extremely effective to reduce the phosphorus contained in the tungsten, strictly speaking, abnormal grain growth still occurred, and additional improvement was required.

Generally speaking, a sintered compact is prepared via HIP in order to attain higher density and higher strength of the tungsten target, but since abnormal grain growth at this stage causes the problem of processing defects in the post-processing, there is a task of further reducing abnormal grain growth.

50 **[0010]** Other than the foregoing Patent Documents, Patent Document 9 describes adjusting the grain size and crystal structure via rotary forging, but the object of Patent Document 9 is not to prevent the abnormal grain growth of tungsten, and under the current circumstances there are no specific means for preventing abnormal grain growth.

Patent Document 1: JP 3086447 B

55 Patent Document 2: JP 2001-098364 A

Patent Document 3: WO 2009/147900

Patent Document 4: JP 2005-171389 A

Patent Document 5: JP 2007-314883 A

Patent Document 6: JP H05-093267 A  
 Patent Document 7: JP 2001-335923 A  
 Patent Document 8: JP H07-076771 A  
 Patent Document 9: JP 2012-180599 A

## SUMMARY OF INVENTION

### [Technical Problem]

**[0011]** In light of the foregoing points, there is a problem in that abnormal grain growth and deterioration in target strength occur during the production stage of a tungsten sintered compact sputtering target and consequently deteriorate the product yield. An object of this invention is to inhibit abnormal grain growth of tungsten and deterioration in target strength.

### [Solution to Problem]

**[0012]** In order to achieve the foregoing object, the present inventors provide the following invention.

- 1) A tungsten sintered compact sputtering target containing iron as an impurity in an amount of 0.8 wtppm or less, and remainder being tungsten and other unavoidable impurities, wherein a range of iron concentration in a target structure is within a range of  $\pm 0.1$  wtppm of an average concentration.
- 2) The tungsten sintered compact sputtering target according to 1) above, wherein a relative density of the target is 99% or higher, an average crystal grain size is 50  $\mu\text{m}$  or less, and a crystal grain size range is 5 to 200  $\mu\text{m}$ .
- 3) The tungsten sintered compact sputtering target according to 1) or 2) above, wherein a purity of tungsten excluding gas components is 5N (99.999%) or higher.
- 4) The tungsten sintered compact sputtering target according to 3) above, wherein contents of oxygen and carbon are respectively 50 wtppm or less.

### [Advantageous Effects of Invention]

**[0013]** As a result of producing a tungsten sintered compact sputtering target containing iron as an impurity in an amount of 0.8 wtppm or less, and remainder being tungsten and other unavoidable impurities, the present invention yields a superior effect of being able to inhibit abnormal grain growth and deterioration in target strength during the production stage of the tungsten sintered compact sputtering target. Consequently, the present invention yields an effect of being able to reduce the problem of processing defects that occur in the post-processing, and improve the product yield.

## BRIEF DESCRIPTION OF DRAWINGS

**[0014]** [Fig. 1] It is a schematic explanatory diagram showing 17 measurement points in total for iron concentration measurement.

## DESCRIPTION OF EMBODIMENTS

**[0015]** The tungsten sintered compact sputtering target of the present invention is unique in that it contains iron as an impurity in an amount of 0.8 wtppm or less, and remainder being tungsten and other unavoidable impurities. The iron content is limited to be an extremely small amount, and this is extremely important in the present invention. It is thereby possible to yield the effect of being able to inhibit abnormal grain growth and deterioration in target strength, and effectively prevent the occurrence of defects in subsequent processing. It is even more effective to cause the content of iron as an impurity to be 0.5 wtppm or less.

**[0016]** It is particularly effective to cause the relative density to be 99% or higher, the average crystal grain size to be 50  $\mu\text{m}$  or less, and the crystal grain size to be 5 to 200  $\mu\text{m}$  in order to maintain the strength of the tungsten sintered compact sputtering target.

**[0017]** The tungsten sintered compact sputtering target of the present invention preferably has a purity of 5N (99.999%) or higher. This purity level is effective in maintaining the strength of the tungsten sintered compact sputtering target. With the tungsten sintered compact sputtering target of the present invention, the range of iron concentration in the target structure needs to be within the range of  $\pm 0.1$  wtppm of the average concentration.

**[0018]** This is because, even if the content of iron as an impurity contained in the tungsten is caused to be 0.8 wtppm or less in the target, when there is segregation in the target structure, the segregation spot in the structure tends to

become the source of abnormal grain growth. Inhibition of the foregoing variation is effective in reducing the foregoing problems.

In addition, it is effective to cause the contents of hydrogen, carbon, nitrogen, oxygen and sulfur, which are gas components, to be respectively 50 wtppm or less in order to similarly maintain the strength of the tungsten sintered compact sputtering target.

**[0019]** When producing a tungsten sintered compact sputtering target, a tungsten raw material powder in which the content of iron as an impurity is 0.8 wtppm or less is used, and sufficiently mixed so that variation in the Fe content becomes small. This mixing may be performed, for example, using a ball mill or a V-type mixer. The iron concentration in the raw material powder should be suppressed to be 0.8 wtppm or less based on ICP analysis. In particular, preferably, the raw material powder is divided into 25 portions, each portion is subject to analysis, and the range of variation in the analytical value of iron is within the range of  $\pm 0.1$  wtppm of the average concentration.

**[0020]** It is thereby possible to cause the range of iron concentration in the target structure to be within the range of  $\pm 0.1$  wtppm of the average concentration. Since the content of Fe in the tungsten is low, variation in the Fe content can be inhibited and uniformized mainly by sufficiently mixing the raw material powder. Specifically, it is effective to measure the iron concentration based on the following conditions and arbitrarily set the compatible production conditions so as to control the variation in the stationary iron concentration in the tungsten target structure. In this respect, a clear process of measuring and controlling the variation of the iron concentration in the target structure is important.

**[0021]** With regard to the measurement of iron concentration in the target, for instance, in the case of a disk-shaped tungsten target, measurement is performed at 17 points (1 point is the center). In other words, as shown in Fig. 1, the iron concentration is measured at a total of 17 points; specifically, the center, 8 equally-spaced points on  $1/2 R$  (radius), and 8 equally-spaced points on the outer periphery.

**[0022]** The tungsten raw material powder adjusted as described above of which average grain size is roughly  $1 \mu\text{m}$  is filled in a carbon die, hot pressed at a temperature of 1500 to 1800°C, and thereafter subject to HIP treatment at a temperature of 1600 to 1850°C and a pressure of 1700 to 1800 kgf for 3 to 4 hours to produce the tungsten sintered compact sputtering target of the present invention. The foregoing HIP treatment conditions are conditions that are normally adopted, and HIP treatment may also be performed outside the range of the foregoing conditions as needed. It is thereby possible to achieve an average grain size of 20 to 30  $\mu\text{m}$  and a relative density of 99%.

**[0023]** Nevertheless, since the tungsten sintered compact sputtering target is produced using a carbon die, a large amount of carbon (C) will be included, as an impurity, within the sintered compact target. In the foregoing case, as the C content increases, the specific resistance of the tungsten film after the sputter deposition tends to increase. Thus, the content of carbon as a gas component should be reduced to 50 wtppm or less.

**[0024]** Since oxygen bonds with the impurities contained in the tungsten to form oxides, it is desirable to simultaneously reduce oxygen. Moreover, the gas component of oxygen also reacts with tungsten to also form an oxide. Thus, the oxygen content should be reduced to 50 wtppm or less.

Moreover, the total impurity concentration excluding gas components is preferably 4 wtppm or less, more preferably 3 wtppm or less, and most preferably 2 ppm or less.

**[0025]** Since impurities excluding gas components get mixed into the LSI wiring material during sputter deposition and cause the deterioration in the function of the tungsten wiring, it could be said that such impurities should be reduced as much as possible. Moreover, the foregoing reduction of carbon, oxygen and iron additionally yields the effect of uniformizing the structure and inhibiting the generation of cracks and fractures of the target. In addition, this tungsten sintered compact sputtering target can be used for depositing a film on a substrate, and is effective for producing a semiconductor device.

**[0026]** During sintering, it is effective to perform hot press (HP) at a temperature exceeding 1500°C. Moreover, after the hot press, HIP treatment can be performed at a temperature exceeding 1600°C in order to further improve the density. Moreover, it is possible to provide a tungsten sintered compact sputtering target having a relative density of 99% or higher, and even 99.5% or higher. Improvement of density is favorable since it can increase the strength of the target.

**[0027]** Accordingly, the target structure becomes uniform in the radial direction and thickness direction of the target, and the strength of the target is sufficient. Thereby, the problem of the target cracking during operation or use can be eliminated, and it becomes possible to improve the production yield of the target.

As described above, the content of iron as an impurity can be reduced to be 0.8 wtppm or less, and even 0.5 wtppm or less. It is thereby possible to yield the effect of being able to inhibit abnormal grain growth and deterioration in target strength, and effectively prevent the occurrence of defects in the subsequent processing.

**[0028]** As described above, the sputtering target of the present invention can maintain the density at a high level, reduce holes and cause the crystal grains to become refined to achieve the uniform and smooth sputtered surface of the target. Thus, the sputtering target of the present invention yields the effect of being able to reduce the generation of particles and nodules during the sputtering process and additionally extend the target life, and additionally yields the effect of being able to reduce the variation in quality and improve mass productivity.

[Examples]

**[0029]** The present invention is now explained based on the Examples and Comparative Examples. These Examples are merely illustrative, and the present invention shall in no way be limited thereby. In other words, the present invention is only limited by the scope of claims and, as a matter of course, shall cover various modifications and embodiments other than Examples below.

(Example 1)

**[0030]** A refined W (tungsten) powder was sufficiently mixed to prepare a W powder to attain an average Fe concentration of 0.4 wtpm and a range of Fe concentration of 0.3 to 0.5 wtpm (25-portion measurement), and the prepared W powder was hot pressed at a temperature of 1800°C and a pressure of 200 kgf.

Subsequently, the obtained product was subject to HIP under the following conditions; namely, a temperature of 1700°C and a pressure of 1800 kgf for 3.0 hours. The thus produced tungsten target was free from grain growth that would lead to a processing defect.

With this sputtering target, it was possible to maintain an average Fe concentration of 0.4 wtpm, and a range of Fe concentration of 0.3 to 0.5 wtpm (17-point measurement).

**[0031]** The P content was 0.5 wtpm or less, the average crystal grain size was 20  $\mu\text{m}$ , the average crystal grain size range was 15 to 25  $\mu\text{m}$ , the total impurity concentration was 1.001 wtpm, the content of oxygen as a gas component was 30 wtpm, and the carbon content was 20 wtpm. This tungsten target satisfied the conditions of the present invention. In addition, the target was free from abnormal grain growth and deterioration in target strength, and furthermore the subsequent workability was favorable.

Note that the grain size was measured by polishing and etching W and observing the structure with an optical microscope. In other words, on the structure photograph of one microscopic field, two straight lines were drawn both in the vertical direction and in the horizontal direction, the number of grain boundaries intersecting with these lines was counted, and the grain size was determined by dividing the total length of these four lines by the number of grain boundaries intersecting with these lines (crosscut method). The grain size of the largest grain and the grain size of the smallest grain in one microscopic field of the structure were measured, and a range of the result values is defined as the range. The measurement locations were 17 points. The same method was adopted for measuring the grain size in the following Examples and Comparative Examples.

**[0032]** In particular, the crystal grain size considerably affects the workability. For example, a normal turning process is enabled when the average crystal grain size is small; but if the grain size is large, grains tend to fall off, and there is a problem in that a smooth surface cannot be obtained in the structure after being subject to the turning process. When numerous grains fall off, that location may become the source of cracks, and there are cases where the process itself cannot be performed. The same applies to the following Examples and Comparative Examples. The results are shown in Table 1.

[Table 1]

	Average Fe concentration (wtppm)	Range of Fe concentration (wtppm)	HIP conditions			Average crystal grain size ( $\mu\text{m}$ )	Range of average crystal grain size ( $\mu\text{m}$ )	Total impurity concentration (wtppm)	Oxygen content (wtppm)	Carbon content (wtppm)	Workability
			Temperature ( $^{\circ}\text{C}$ )	Pressure (kgf)	Holding time (hr)						
Example 1	0.4	0.3 to 0.5	1700	1800	3.0	20	15-25	1.001	30	20	○
Example 2	0.7	0.6 to 0.8	1750	1700	4.0	25	20-30	1.003	20	20	○
Example 3	0.8	0.7 to 0.9	1750	1700	4.0	30	20-35	1.013	20	20	○
Example 4	0.7	0.6 to 0.8	1770	1770	2.0	50	5-200	1.003	30	30	○
Comparative Example 1	1.0	0.9 to 1.1	1700	1800	8.0	800	600-1000	1.005	30	30	×
Comparative Example 2	0.4	0.2 to 0.8	1750	1700	6.0	500	400-650	1.002	30	20	×
Comparative Example 3	0.7	0.4 to 0.9	1700	1400	3.0	700	500-850	1.001	20	20	×
Comparative Example 4	1.0	0.9 to 1.1	1750	1500	4.0	900	800-1000	5.201	40	40	×
Comparative Example 5	0.7	0.4 to 0.9	1600	1800	3.0	900	850-950	4.606	120	20	×
Comparative Example 6	0.4	0.2 to 0.8	1500	1700	4.0	800	700-900	4.293	30	160	×

(Example 2)

**[0033]** A refined W powder was sufficiently mixed to prepare a W powder to attain an average Fe concentration of 0.7 wtppm and a range of Fe concentration of 0.6 to 0.8 wtppm (25-portion measurement), and the prepared W powder was hot pressed at a temperature of 1800°C and a pressure of 200 kgf.

Subsequently, the obtained product was subject to HIP under the following conditions; namely, a temperature of 1750°C and a pressure of 1700 kgf for 4.0 hours. The thus produced tungsten target was free from grain growth that would lead to a processing defect.

**[0034]** With this sputtering target, it was possible to maintain an average Fe concentration of 0.7 wtppm, and a range of Fe concentration of 0.6 to 0.8 wtppm (17-point measurement).

The P content was 0.5 wtppm or less, the average crystal grain size was 25  $\mu\text{m}$ , the average crystal grain size range was 20 to 30  $\mu\text{m}$ , the total impurity concentration was 1.003 wtppm, the content of oxygen as a gas component was 20 wtppm, and the carbon content was 20 wtppm. This tungsten target satisfied the conditions of the present invention. In addition, the target was free from abnormal grain growth and deterioration in target strength, and furthermore the subsequent workability was favorable. The results are similarly shown in Table 1.

(Example 3)

**[0035]** A refined W powder was sufficiently mixed to prepare a W powder to attain an average Fe concentration of 0.8 wtppm and a range of Fe concentration of 0.7 to 0.9 wtppm (25-portion measurement), and the prepared W powder was hot pressed at a temperature of 1800°C and a pressure of 200 kgf.

Subsequently, the obtained product was subject to HIP under the following conditions; namely, a temperature of 1750°C and a pressure of 1700 kgf for 4.0 hours. The thus produced tungsten target was free from grain growth that would lead to a processing defect.

**[0036]** With this sputtering target, it was possible to maintain an average Fe concentration of 0.8 wtppm, and a range of Fe concentration of 0.7 to 0.9 wtppm (17-point measurement).

The P content was 0.5 wtppm or less, the average crystal grain size was 30  $\mu\text{m}$ , the average crystal grain size range was 20 to 35  $\mu\text{m}$ , the total impurity concentration was 1.013 wtppm, the content of oxygen as a gas component was 20 wtppm, and the carbon content was 20 wtppm. This tungsten target satisfied the conditions of the present invention. In addition, the target was free from abnormal grain growth and deterioration in target strength, and furthermore the subsequent workability was favorable. The results are similarly shown in Table 1.

(Example 4)

**[0037]** A refined W powder was sufficiently mixed to prepare a W powder to attain an average Fe concentration of 0.7 wtppm and a range of Fe concentration of 0.6 to 0.8 wtppm (25-portion measurement), and the prepared W powder was hot pressed at a temperature of 1800°C and a pressure of 200 kgf.

Subsequently, the obtained product was subject to HIP under the following conditions; namely, a temperature of 1770°C and a pressure of 1770 kgf for 2.0 hours. The thus produced tungsten target was free from grain growth that would lead to a processing defect.

**[0038]** With this sputtering target, it was possible to maintain an average Fe concentration of 0.7 wtppm, and a range of Fe concentration of 0.6 to 0.8 wtppm (17-point measurement).

The P content was 0.5 wtppm or less, the average crystal grain size was 50  $\mu\text{m}$ , the average crystal grain size range was 5 to 200  $\mu\text{m}$ , the total impurity concentration was 1.003 wtppm, the content of oxygen as a gas component was 30 wtppm, and the carbon content was 30 wtppm. This tungsten target satisfied the conditions of the present invention. In addition, the target was free from abnormal grain growth and deterioration in target strength, and furthermore the subsequent workability was favorable. The results are similarly shown in Table 1.

(Comparative Example 1)

**[0039]** A W powder having an average Fe concentration of 1.0 wtppm was prepared. The range of Fe concentration was 0.9 to 1.1 wtppm (25-portion measurement). The prepared W powder was hot pressed at a temperature of 1800°C and a pressure of 200 kgf, and the obtained product was subsequently subject to HIP under the following conditions; namely, a temperature of 1700°C and a pressure of 1800 kgf for 8.0 hours. Consequently, grain growth that would lead to a processing defect occurred.

**[0040]** With this sputtering target, the average Fe concentration was 1.0 wtppm, and the range of Fe concentration was 0.9 to 1.1 wtppm (17-point measurement). Thus this sputtering target failed to satisfy the conditions of the present invention.

The P content was 0.5 wtppm or less, the average crystal grain size was 800  $\mu\text{m}$ , the average crystal grain size range was 600 to 1000  $\mu\text{m}$ , the total impurity concentration was 1.005 wtppm, the content of oxygen as a gas component was 30 wtppm, and the carbon content was 30 wtppm. From a comprehensive perspective, this sputtering target failed to satisfy the conditions of the present invention. In particular, the average crystal grain size and the average crystal grain size range had considerably deviated from the conditions of the present invention. Consequently, abnormal grain growth was observed in the target, the strength of the target deteriorated, and the subsequent workability was inferior. The results are similarly shown in Table 1.

(Comparative Example 2)

**[0041]** A W powder having an average Fe concentration of 0.4 wtppm was prepared. The range of Fe concentration was 0.2 to 0.8 wtppm (25-portion measurement). The prepared W powder was hot pressed at a temperature of 1800°C and a pressure of 200 kgf, and the obtained product was subsequently subject to HIP under the following conditions; namely, a temperature of 1750°C and a pressure of 1700 kgf for 6.0 hours. Consequently, grain growth that would lead to a processing defect occurred.

**[0042]** With this sputtering target, the average Fe concentration was 0.4 wtppm, the range of Fe concentration was 0.2 to 0.8 wtppm (17-point measurement), and variation in the maximum-minimum range was considerable and failed to satisfy the conditions of the present invention.

The P content was 0.5 wtppm or less, the average crystal grain size was 500  $\mu\text{m}$ , the average crystal grain size range was 400 to 650  $\mu\text{m}$ , the total impurity concentration was 1.002 wtppm, the content of oxygen as a gas component was 30 wtppm, and the carbon content was 20 wtppm. From a comprehensive perspective, this sputtering target failed to satisfy the conditions of the present invention. In particular, the average crystal grain size and the average crystal grain size range had considerably deviated from the conditions of the present invention. Consequently, abnormal grain growth was observed in the target, the strength of the target deteriorated, and the subsequent workability was inferior. The results are similarly shown in Table 1.

(Comparative Example 3)

**[0043]** A W powder having an average Fe concentration of 0.7 wtppm was prepared.

The range of Fe concentration was 0.4 to 0.9 wtppm (25-portion measurement). The prepared W powder was hot pressed at a temperature of 1800°C and a pressure of 200 kgf, and the obtained product was subsequently subject to HIP under the following conditions; namely, a temperature of 1700°C and a pressure of 1400 kgf for 3.0 hours. Consequently, grain growth that would lead to a processing defect occurred.

**[0044]** With this sputtering target, the average Fe concentration was 0.7 wtppm, the range of Fe concentration was 0.4 to 0.9 wtppm (17-point measurement), and variation in the maximum-minimum range was considerable and failed to satisfy the conditions of the present invention.

The P content was 0.5 wtppm or less, the average crystal grain size was 700  $\mu\text{m}$ , the average crystal grain size range was 500 to 850  $\mu\text{m}$ , the total impurity concentration was 1.001 wtppm, the content of oxygen as a gas component was 20 wtppm, and the carbon content was 20 wtppm. From a comprehensive perspective, this sputtering target failed to satisfy the conditions of the present invention. In particular, the average crystal grain size and the average crystal grain size range had considerably deviated from the conditions of the present invention. Consequently, abnormal grain growth was observed in the target, the strength of the target deteriorated, and the subsequent workability was inferior. The results are similarly shown in Table 1.

(Comparative Example 4)

**[0045]** A W powder having an average Fe concentration of 1.0 wtppm was prepared. The range of Fe concentration was 0.9 to 1.1 wtppm (25-portion measurement). The prepared W powder was hot pressed at a temperature of 1800°C and a pressure of 200 kgf, and the obtained product was subsequently subject to HIP under the following conditions; namely, a temperature of 1750°C and a pressure of 1500 kgf for 4.0 hours. Consequently, grain growth that would lead to a processing defect occurred.

**[0046]** With this sputtering target, the average Fe concentration was 1.0 wtppm, and the range of Fe concentration was 0.9 to 1.1 wtppm (17-point measurement). The average Fe concentration failed to satisfy the condition of the present invention.

The P content was 0.5 wtppm or less, the average crystal grain size was 900  $\mu\text{m}$ , the average crystal grain size range was 800 to 1000  $\mu\text{m}$ , the total impurity concentration was 5.201 wtppm, the content of oxygen as a gas component was 40 wtppm, and the carbon content was 40 wtppm. From a comprehensive perspective, this sputtering target failed to satisfy the conditions of the present invention. In particular, the average crystal grain size and the average crystal grain



size range had considerably deviated from the conditions of the present invention. Consequently, abnormal grain growth was observed in the target, the strength of the target deteriorated, and the subsequent workability was inferior. The results are similarly shown in Table 1.

5 (Comparative Example 5)

[0047] A W powder having an average Fe concentration of 0.7 wtppm was prepared. The range of Fe concentration was 0.4 to 0.9 wtppm (25-portion measurement). The prepared W powder was hot pressed at a temperature of 1800°C and a pressure of 200 kgf, and the obtained product was subsequently subject to HIP under the following conditions; namely, a temperature of 1600°C and a pressure of 1800 kgf for 3.0 hours. Consequently, grain growth that would lead to a processing defect occurred.

[0048] With this sputtering target, the average Fe concentration was 0.7 wtppm, the range of Fe concentration was 0.4 to 0.9 wtppm (17-point measurement), and variation in the range of Fe concentration was considerable and failed to satisfy the condition of the present invention.

15 The P content was 0.5 wtppm or less, the average crystal grain size was 900 μm, the average crystal grain size range was 850 to 950 μm, the total impurity concentration was 4.606 wtppm, the content of oxygen as a gas component was 120 wtppm, and the carbon content was 20 wtppm. From a comprehensive perspective, this sputtering target failed to satisfy the conditions of the present invention. In particular, the average crystal grain size, the average crystal grain size range and the oxygen content had considerably deviated from the conditions of the present invention. Consequently, abnormal grain growth was observed in the target, the strength of the target deteriorated, and the subsequent workability was inferior. The results are similarly shown in Table 1.

(Comparative Example 6)

25 [0049] A W powder having an average Fe concentration of 0.4 wtppm was prepared. The range of Fe concentration was 0.2 to 0.8 wtppm (25-portion measurement). The prepared W powder was hot pressed at a temperature of 1800°C and a pressure of 200 kgf, and the obtained product was subsequently subject to HIP under the following conditions; namely, a temperature of 1500°C and a pressure of 1700 kgf for 4.0 hours. Consequently, grain growth that would lead to a processing defect occurred.

30 [0050] With this sputtering target, the average Fe concentration was 0.4 wtppm, the range of Fe concentration was 0.2 to 0.8 wtppm (17-point measurement), and variation in the range of Fe concentration was considerable and failed to satisfy the condition of the present invention.

The P content was 0.5 wtppm or less, the average crystal grain size was 800 μm, the average crystal grain size range was 700 to 900 μm, the total impurity concentration was 4.293 wtppm, the content of oxygen as a gas component was 30 wtppm, and the carbon content was 160 wtppm. From a comprehensive perspective, this sputtering target failed to satisfy the conditions of the present invention. In particular, the average crystal grain size, the average crystal grain size range and the carbon content had considerably deviated from the conditions of the present invention. Consequently, abnormal grain growth was observed in the target, the strength of the target deteriorated, and the subsequent workability was inferior. The results are similarly shown in Table 1.

40 INDUSTRIAL APPLICABILITY

[0051] The present invention provides a tungsten sintered compact sputtering target containing iron as an impurity contained in tungsten in an amount of 0.8 wtppm or less, and remainder being tungsten and other unavoidable impurities. The present invention is thereby possible to yield a superior effect of being able to inhibit abnormal grain growth and deterioration in target strength during the production stage of the tungsten sintered compact sputtering target. Consequently, the present invention yields an effect of being able to reduce the problem of processing defects that occur in the post-processing, and improve the product yield.

50 Moreover, by depositing a film using this tungsten sintered compact sputtering target, the deposited tungsten film yields a superior effect of being able to stably reduce electrical resistivity. Accordingly, the tungsten sintered compact sputtering target of the present invention is effective in forming an LSI wiring film.

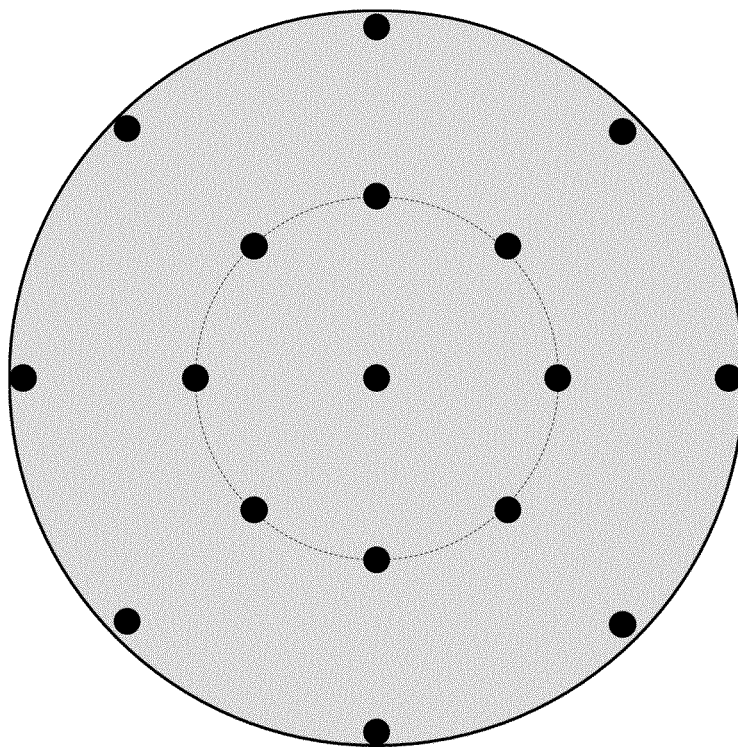
Claims

- 55 1. A tungsten sintered compact sputtering target containing iron as an impurity in an amount of 0.8 wtppm or less, and remainder being tungsten and other unavoidable impurities, wherein a range of iron concentration in a target structure is within a range of  $\pm 0.1$  wtppm of an average concentration.

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2. The tungsten sintered compact sputtering target according to claim 1,  
wherein a relative density of the target is 99% or higher, an average crystal grain size is 50  $\mu\text{m}$  or less, and a crystal  
grain size range is 5 to 200  $\mu\text{m}$ .
3. The tungsten sintered compact sputtering target according to claim 1 or  
claim 2, wherein a purity of tungsten excluding gas components is 5N (99.999%) or higher.
4. The tungsten sintered compact sputtering target according to claim 3,  
wherein contents of oxygen and carbon are respectively 50 wtpm or less.

[Fig. 1]



## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2014/057646

## A. CLASSIFICATION OF SUBJECT MATTER

C23C14/34(2006.01)i, C22C27/04(2006.01)i, H01L21/28(2006.01)i, H01L21/285(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C23C14/00-14/58, C22C27/04, H01L21/28, H01L21/285, B22F1/00, B22F3/12-3/16

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2014  
Kokai Jitsuyo Shinan Koho 1971-2014 Toroku Jitsuyo Shinan Koho 1994-2014

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

JSTPlus(JDreamIII)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2007/0243095 A1 (TOSOH SMD, INC.), 18 October 2007 (18.10.2007), claims 1 to 21; paragraphs [0006] to [0024] & WO 2006/001976 A2	1-4
Y	JP 2001-335923 A (Toshiba Corp.), 07 December 2001 (07.12.2001), claims 1 to 11; paragraphs [0004] to [0006], [0014] to [0016], [0024] to [0025], [0029], [0033]; fig. 1 (Family: none)	1-4

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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Date of the actual completion of the international search  
12 June, 2014 (12.06.14)

Date of mailing of the international search report  
24 June, 2014 (24.06.14)

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## INTERNATIONAL SEARCH REPORT

International application No.

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## C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 2009/147900 A1 (Nippon Mining & Metals Co., Ltd.), 10 December 2009 (10.12.2009), claims 3, 4; paragraphs [0017] to [0018], [0021], [0024] & JP 5243541 B & US 2011/0094879 A1 & EP 2284289 A1 & CN 102046822 A & KR 10-2010-0139152 A & TW 201005113 A	2, 4
A	JP 7-76771 A (Japan Energy Corp.), 20 March 1995 (20.03.1995), claims 1, 2; paragraphs [0011], [0016] to [0019] (Family: none)	1-4
A	Yoshiharu FUKAZAWA et al., "High-purity metal sputtering targets", Toshiba Review, vol.43, no.9, 1988, pages 761 to 764	1-4
A	M. SUZUKI, DEVELOPMENT OF REFRACTORY METALS AND SILICIDES TARGETS, AND THEIR CHARACTERISTICS, TUNGSTEN AND OTHER REFRACTORY METALS FOR VLSI APPLICATIONS II, Materials Research Society, 1987, P339-345, ISSN 0886-7860	1-4
A	JP 8-250427 A (Central Glass Co., Ltd.), 27 September 1996 (27.09.1996), paragraphs [0029] to [0034] (Family: none)	1-4

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**REFERENCES CITED IN THE DESCRIPTION**

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